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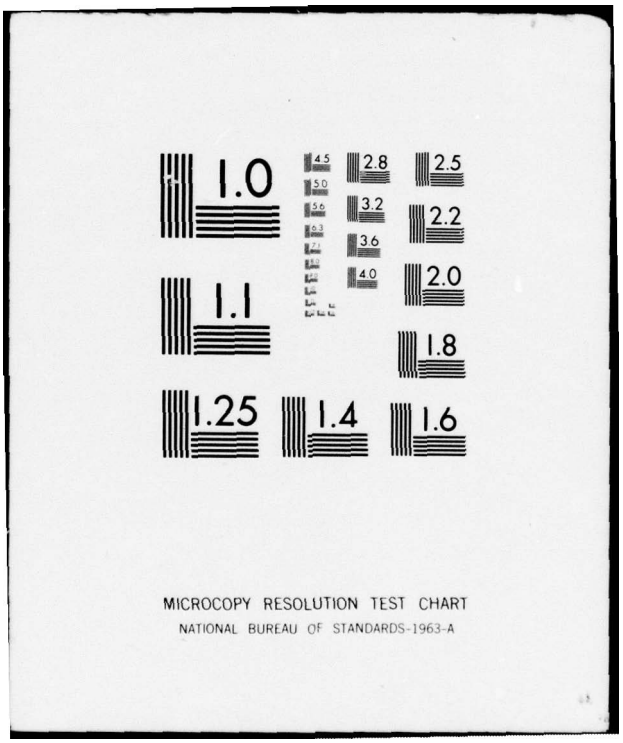
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(6) THE INFLUENCE OF EXTRUSION TEMPERATURE ON THE PROPERTIES OF ULTRA-DRAWN POLYETHYLENE,

by

(10) Shunji Kojima and Roger S. Porter
Polymer Science and Engineering
Materials Research Laboratory
University of Massachusetts
Amherst, Massachusetts 01003

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THE INFLUENCE OF EXTRUSION TEMPERATURE ON THE PROPERTIES OF ULTRA-DRAWN POLYETHYLENE

Shunji Kojima* and Roger S. Porter
Polymer Science and Engineering
Materials Research Laboratory
University of Massachusetts
Amherst, Massachusetts

Abstract

In recent years, many studies have been made in this and other laboratories on the solid state extrusion of thermoplastics. The influence of extrusion conditions on the properties of the resultant fibers and films has not, however, been well examined and understood. A systematic study has thus been made to evaluate the influence of conditions for the extrusion of high density polyethylene. In this paper, extrusion temperature is the principal variable of evaluation. The temperature range for crystal formation prior to extrusion was held to a narrow range (132-136^{o}C). Related variables of molecular weight and draw ratio are discussed in detail in related studies.

*On leave from The Composite Research and Development Center of Toyo Seikan and Toyo Kohan Companies, 22-4, Okazawa, Hodogaya, Yokohama, Japan

Introduction

Many efforts have been made to study the deformation and morphology of polyethylene (1-3) and to make high-strength and/or high-modulus materials from a variety of polymers (4-39). Cold drawing (4-7), quench-rolling (8), hydrostatic (9-12) and direct (13-37) extrusion in the solid state have been the main methods employed.

Among the first investigation of solid state extrusion of high density polyethylene, HDPE, was by Southern in this laboratory (25) and by the research group headed by Professor Ward at Leeds University, England, and by Professor Takayanagi at Kyushu University in Japan (13). Many studies have been made on the extrusion process, on the resultant physical, mechanical and morphological properties, and on the use of the resultant fibers (13-37). The extrusion techniques has also been improved, and, in our hands, a continuous, steady-state extrusion has been developed for draw ratios to over ten (37).

In the perspective of prior work, it appears important to study the influence of extrusion conditions on the properties of the resultant fibers. Takayanagi (28) has reported on the influence of temperature for the extrusion of polypropylene. However, their techniques are quite different from ours. Capiati, et al. (41), recently reported on the influence of extrusion temperature and pressure for the extrusion of polyethylene. His results on the influence of extrusion temperature are meaningful but incomplete. This is because morphology perfection is influenced by both the temperature of crystal formation and the temperature of extrusion.

In the present study, the influence of temperature on extrusion over a relatively high and wide range is examined for its effect on the properties of the resultant HDPE fibers. The HDPE was crystallized in an Instron rheometer from the melt and then extruded at relatively high temperatures, which is the principal difference between our experiments and the excellent studies of Takayanagi.

Experimental

Two molecular weights of high density polyethylene (DuPont Alathon 7050 and 7026) were used. The weight average molecular weights of these samples are 56,000 and 135,000, respectively. The molecular weight and the distribution of molecular weight of these samples have been published (36).

For fiber preparation, three different brass capillaries were used with Teflon coating. All capillaries had 20 degree entrance angles but different draw ratios. The extrusion or draw ratios of the capillaries are calculated from the reduction ratio of cross-sectional areas. Polymer that does not pass the full entrance region is exposed to a draw ratio less than the maximum which is calculated from reservoir and capillary radii only. Thus, the actual draw ratio was calculated when steady-state extrusion was not achieved. The actual draw ratio and maximum draw ratio are shown in Table 1.

The HDPE samples were melted at 180^{o}C in an Instron rheometer and then cooled to the following two specific crystallization temperatures - 132^{o}C for Alathon 7050 and 136^{o}C for Alathon 7026.

The pressure was then increased to 2400 atms at the chosen constant temperature in the sealed rheometer. With crystallization complete, the temperature was changed to the extrusion temperature and the capillary exit unblocked. Extrusion was initiated at a constant pressure of 2400 atms.

The first 20 centimeters of each fiber were not used for the testing because the properties depend on the location along the fiber. This will be discussed later (38).

Young's modulus and tensile strength were determined using an Instron tensile tester. For the determination of modulus, an extensometer was also employed. The strain rates employed were 0.01 and 0.1 $min.^{-1}$ for modulus and strength determination, respectively, and moduli were determined below 0.2% strain.

The peak melting point and degree of crystallinity were determined using a differential scanning calorimeter (Perkin-Elmer DSC-1B) at a heating rate of $10^{o}C/min$. The peak melting point was corrected using an indium standard and the degree of crystallinity was calculated from the area under melting peaks. The heat of fusion for polyethylene and indium used for the calculation was 69.2 and 6.75 cal/g, respectively.

Results and Discussion

Table 2 shows the influence of extrusion temperature on the degree of crystallinity and peak melting point of resultant fibers. It is clearly shown that the degree of crystallinity increases with extrusion temperature and peak melting point stays constant. This may be due to the effect of annealing in the capillary itself.

It is well known (1) that the higher the crystal formation temperature, the thicker are the lamellae formed. In the present case, the polymer in the capillary cone region is under deformation and the deformation rate is a maximum at the cone exit, i.e. at the capillary entrance. After entering the capillary region, the polymer is not extended further and annealing occurs, which involves stress relaxation and crystal growth. It is, therefore, necessary to consider the effect of both deformation and annealing to discuss the formed morphology.

It is important to distinguish between deformation which merely changes the shape of the sample and deformation which changes the internal morphological structure. The ratio of the deformation which merely changes the shape of the sample should be greater when the polymer under deformation has greater movability, i.e. at higher temperature. Therefore, we can expect less chain orientation and fewer stressed tie molecules (i.e. a lower content of continuous crystal) for fibers drawn at higher temperature. A continuous crystal is the idealized model state in which all molecules are aligned and extended with chain ends buried randomly in the crystal lattice.

The effect of annealing is also greater at higher temperature, i.e. more chance of stress relaxation and recrystallization. This is a second reason why we can expect less chain orientation and less stressed tie molecules for fibers formed at higher temperature.

The data are consistent with these concepts. The crystals prepared and the fibers formed at higher temperatures exhibit a higher degree of crystallinity consistent with less chance for chain irregularities or stressed amorphous tie molecules; the polymer is thus effectively annealed after drawing. Peak melting point, however, is independent of the extrusion temperature because it does not depend on the amorphous structure. These trends hold for the different draw ratios and different molecular weight samples studied here. Figure 1 shows the influence of extrusion temperature on Young's modulus. Young's modulus increases slightly but significantly with decreasing extrusion temperature. There is a sharp drop in tensile modulus at the highest temperatures corresponding to incipient melting of the fibers on exiting the die.

Young's modulus depends essentially on the number of tie molecules which sustain stress on stretching. This corresponds to the fraction of continuous crystals. It is well known that the modulus of drawn fibers decreases on annealing due to the relaxation of stressed tie molecules. This can occur below the nominal melting point (42). A decrease in modulus with increasing extrusion temperature is likely due to this feature. At the highest extrusion temperatures, the modulus drops

precipitously due to actual filament melting on extrusion. Among the several studies of extrusion temperature in this lab, only one showed Young's modulus to increase with extrusion temperature. The data in the same study as a function of pressure, however, do indicate that Young's modulus decreases with increasing extrusion temperature. The difference is thus likely due to an increase in the degree of undercooling for extrusion due to the enhanced pressure.

Table 2 shows that tensile strength is insensitive to extrusion temperature. Tensile strength is influenced by the number and nature of weak points for stress concentration. The slippage between fibrils may, in this case, be the main reason for fracture as demonstrated by Capiati (41). These results thus suggest that the interaction between fibrils, following the tensile strength, is independent of extrusion temperature.

The optical clarity of fibers decreases at the highest extrusion temperatures. This suggests the formation of lamellae with perhaps spherulitic structure. This structure cannot be formed without an annealing or recrystallization process in highly-drawn fibers since related tests indicate that extrusion without annealing totally destroys the original spherulites.

These results show that the higher extrusion temperatures give fibers of higher crystallinity and lower modulus; the tensile strength and peak melting point are not affected by temperature over the range tested. It should be noted that extrusion rate decreases with decreasing extrusion temperature making it difficult to extrude

at lower temperatures than examined here. Exploration of extrusion is being explored at lower temperatures (43).

The molecular weight study by Perkins, et al. (36), shows a difference of peak melting point between extruded Alathon 7050 and 7026 of about 4°C. Polymer molecular weight is thus an important factor which influences both extrusion rate and properties of resultant fibers. A lower extrusion rate and increased tensile properties are expected for higher molecular weight samples.

At 5-10°C below the fiber melting point, the extrusion rate is reasonably fast, samples are defect free, and Young's modulus increases only modestly with further decrease in extrusion temperature. Takayanagi (19) has proposed that the best extrusion temperature is between the melting point and α transition temperature. He extruded mainly at lower temperatures near 110°C for high density polyethylene. It is, however, quite difficult to obtain high draw ratio at these temperatures. This is a principal difference between this work and that of Takayanagi.

Conclusions

These new data lead to several conclusions regarding the solid-state extrusion of high density polyethylene:

Lower extrusion temperatures yield filaments of higher Young's modulus with results insensitive to polymer molecular weight for extrusion draw ratios over

a wide range. Peak melting point and tensile strength are invariant for these extrusion conditions.

A convenient extrusion temperature for preparation of high-modulus fibers was determined to be between 5 to 10^{o}C below the melting point of the resultant fibers, dependent on polyethylene molecular weight and draw ratio.

Acknowledgment

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TABLE 1

Draw Ratio for Capillary Entrance Zones

Capillary Draw Ratio		
Calculated Maximum	Actual	Sample Studied
10.7	10.7	After First 10 cm
11.8	11.8	After First 11 cm
35.9	31 ± 4*	Between 20 and 30 cm

*Samples did not pass through full capillary entrance extrusion draw region.

TABLE 2

Influence of Extrusion Temperature on Percent Crystallinity, Peak Melting Point and Tensile Strength

Sample	Draw Ratio	Extrusion Temperature, °C	Crystallinity, %	Peak Melting Point, °C	Tensile Strength x 10^9 /dynes/cm^2
Alathon 7050	10.7	110	81.8	136.5	2.59
(M_w = 59,000)		115	80.0	136.1	2.44
		120	81.7	136.2	2.31
		125	81.6	136.6	2.39
		130	82.2	136.3	2.37
Alathon 7026	11.8	110	78.0	138.0	3.75
(M_w = 147,000)		115	77.1	137.6	3.27
		120	77.5	138.1	3.98
		125	79.5	137.9	3.84
		130	81.4	138.4	3.90
Alathon 7050	31±4	132	86.1	138.0	3.85
(M_w = 59,000)		136	86.3	138.4	4.30

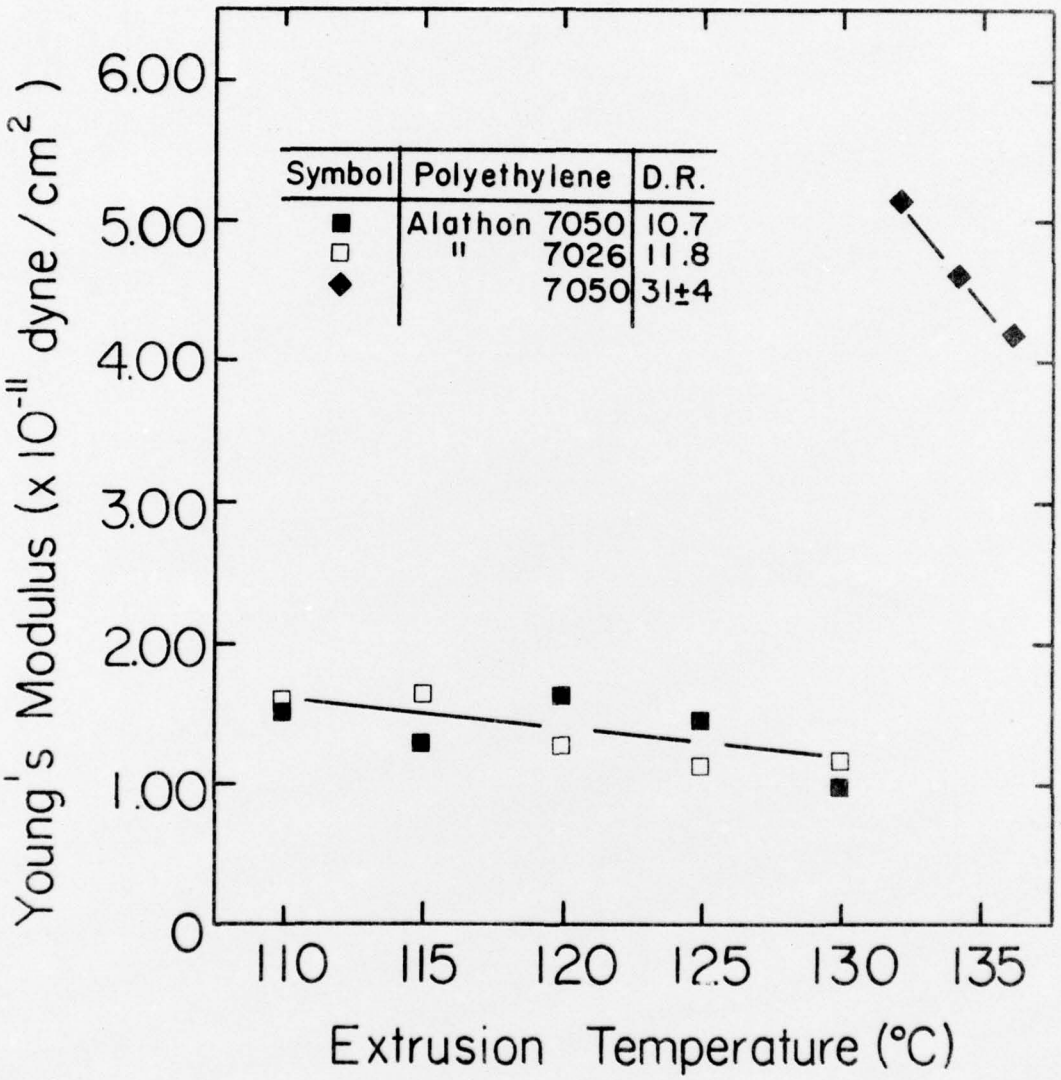

Figure 1

Young's Modulus as a Function of Extrusion Temperature

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In recent years, many studies have been made in this and other laboratories on the solid state extrusion of thermoplastics. The influence of extrusion conditions on the properties of the resultant fibers and films has not, however, been well examined and understood. A systematic study has thus been made to evaluate the influence of conditions for the extrusion of high density polyethylene. In this paper, extrusion temperature is the principal variable of evaluation. The temperature range for crystal formation prior to extrusion was held to a narrow

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range (132-136°C). Related variables of molecular weight and draw ratio are discussed in detail in related studies.

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